IS 5347-9 (1998): Requirements for Orthopaedic Implants, Part 9: Ceramic Materials Based on High Purity Alumina [MHD 2: Orthopaedic Instruments, Implants and Accessories]

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IS 5347 (Part 9) : 1998
ISO 6474 : 1994

भारतीय मानक

अस्थि अन्तर्रोपणों की अपेक्षाएँ

भाग 9 शुद्धतम ऐल्मुना पर आधारित सिरेमिक सामग्री

(पहला पुनरीक्षण)

Indian Standard

REQUIREMENTS FOR ORTHOPAEDIC IMPLANTS

PART 9 CERAMIC MATERIALS BASED ON HIGH PURITY ALUMINA

(First Revision)

ICS 11.040.40

BUREAU OF INDIAN STANDARDS
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July 1998 **Price Group 3**

Orthopaedic Instruments and Accessories Sectional Committee, MHD 2

NATIONAL FOREWORD

This Indian Standard (Part 9) (First Revision) which is identical with ISO 6474 : 1994 'Implants for surgery — Ceramic materials based on high purity alumina' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of Orthopaedic Instruments and Accessories Sectional Committee and approval of the Medical Equipment and Hospital Planning Division Council.

This standard was first published in 1984 as dual number standard. Its first revision has been issued to incorporate the modifications effected in the second edition of ISO 6474 brought out in 1994. In this revised version, the material has been classified in two types that is Type A and Type B. Physical and chemical properties of both the types are given in a tabular form. Details of apparatus used, procedure to be followed, methods of preparation of test pieces have been included for different types of tests.

The text of above mentioned ISO standard has been approved as suitable for publication as Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following :

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their place are listed below along with their degree of equivalence for the editions indicated :

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 468 : 1982	IS 3073 : 1967 Assessment of surface roughness	Technically Equivalent
ISO 3611 : 1978	IS 2967 : 1983 External micrometers (*first revision*)	do
ISO 5017 : 1988	IS 1528 (Part 8) : 1974 Methods of sampling and physical tests for refractory material : Part 8 Determination of apparent porosity (*first revision*)	do
	IS 1528 (Part 9) : 1995 Methods of sampling and physical tests for refractory material : Part 9 Determination of specific gravity and true density (*third revision*)	do

(*Continued on third cover*)

Indian Standard

REQUIREMENTS FOR ORTHOPAEDIC IMPLANTS

PART 9 CERAMIC MATERIALS BASED ON HIGH PURITY ALUMINA

(First Revision)

1 Scope

This International Standard specifies the characteristics of, and corresponding test methods for, a bio-compatible and bio-stable ceramic bone substitute material based on high purity alumina for use as bone spacers, bone replacements and components of orthopaedic joint prostheses.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 468:1982, *Surface roughness — Parameters, their values and general rules for specifying requirements.*

ISO 3611:1978, *Micrometer callipers for external measurement.*

ISO 5017:1988, *Dense shaped refractory products — Determination of bulk density, apparent porosity and true porosity.*

ISO 5436:1985, *Calibration specimens — Stylus instruments — Types, calibration and use of specimens.*

ASTM C573:1986, *Method for chemical analysis of fireclay and high-alumina refractories.*

ASTM E112:1988, *Methods for determining average grain size.*

3 Classification

The material shall be classified as either type A or type B.

Ceramic materials of type A are intended for implants for high load applications (e.g. bearing surfaces of joint replacements) and type B is intended for implants for low load application (e.g. maxillofacial and middle-ear implants).

4 Physical and chemical properties

The properties of type A and type B materials shall be as given in table 1.

5 Test methods

5.1 Bulk density

The bulk density shall be determined in accordance with ISO 5017.

5.2 Chemical composition

The chemical composition shall be determined either in accordance with ASTM C573, or by an equivalent method. In cases of dispute, the method given in ASTM C573 shall be the referee method.

Table 1 — Properties of type A and type B materials

Property	Unit	Requirement		Test method according to subclause
		Type A	Type B	
Bulk density	g/m³	⩾ 3,94	⩾ 3,90	5.1
Chemical composition:				5.2
basic material, Al_2O_3	%	⩾ 99,5		
sintering additive, MgO	%	⩽ 0,3		
limits of impurities, total amount of SiO_2 + CaO + alkali metal oxides	%	⩽ 0,1		
Microstructure:				5.3
mean linear intercept size	µm	⩽ 4,5	⩽ 7,0	
standard deviation	µm	⩽ 2,6	⩽ 3,5	
Average biaxial flexural strength	MPa	⩾ 250	⩾ 150	5.4
Wear resistance[1)]				5.5
wear volume	mm³	⩾ 0,1	not applicable	

1) This test applies only if articulation of ceramic on ceramic is intended.

5.3 Microstructure

5.3.1 Principle

As a means of describing the microstructure, the average grain size is determined by measuring the mean linear intercept size.

5.3.2 Apparatus

5.3.2.1 Grinding and polishing devices to prepare plane and smooth scratch-free surfaces.

5.3.2.2 Furnace capable of maintaining a temperature of 1 500 °C.

5.3.2.3 Light microscope having a magnification of × 500 to × 1 000.

NOTE 1 If the mean grain size is anticipated to be less than 2 µm, a scanning optical microscope may be preferred.

5.3.3 Preparation of test pieces

5.3.3.1 Prepare test pieces of the alumina ceramic by methods representative of the processing techniques to be used for the production of parts for surgery, using the same precursor powder, pressing method and pressure, and firing conditions.

5.3.3.2 Grind one surface of the test piece plane and polish it until the percentage of scratch-free interpretable area is at least 90 %.

5.3.3.3 Etch the test pieces thermally in air at a temperature in the range 1 400 °C to 1 500 °C for 1 h to 4 h.

To increase the optical contrast, the polished and etched surface may be sputter-coated with a thin gold layer.

5.3.4 Procedure

Observe the microstructure using the microscope at a magnification sufficient to delineate grain boundaries clearly. Using either lines drawn on photomicrographs or stage movement, follow the general procedure in ASTM E112 to measure the individual linear intercept sizes of at least 250 grains in total over at least six fields of view on lines sufficiently long to encompass at least 20 grains, taking random orientations of measurement. Calibrate the magnification employed using a certified graticule or grid. Alternatively a calibrated stage micrometer may be used.

5.3.5 Calculation of results

Calculate the mean linear intercept size and the standard deviation from the individual linear intercept sizes.

5.3.6 Test report

The test report shall contain the following information:

a) the identity of the ceramic material, details of the batch number and other codes sufficient to identify the test pieces uniquely;

b) the method of preparing the test pieces surfaces, including details of the grinding and polishing procedure used;

c) the mean linear intercept size and its standard deviation, expressed in micrometres.

5.4 Biaxial flexural strength

5.4.1 Principle

A disc of test material is placed between two coaxial rings of unequal diameter, and a compressive force is applied. The force at fracture of the test disc is recorded and the fracture nominal stress is calculated.

5.4.2 Apparatus

5.4.2.1 Mechanical testing machine suitable for applying a compressive load of at least 5 kN at a nominal loading rate of (500 ± 100) $N \cdot s^{-1}$ and equipped to record the peak force applied to an accuracy of better than 1 %.

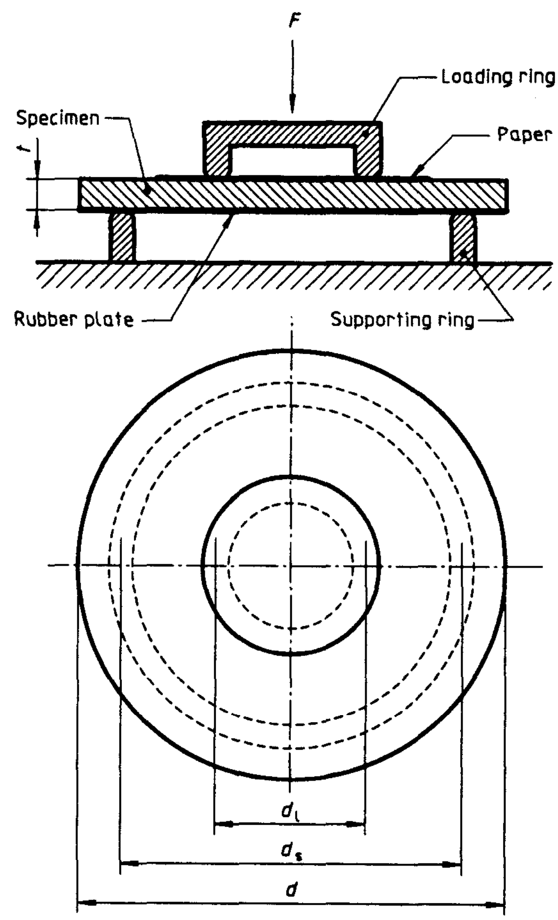
Calibration of the test machine shall be carried out according to agreed procedures, e.g. ASTM E4-83.

5.4.2.2 Test jig comprising unequal diameter loading rings and having a geometry typically as shown in figure 1. The jig shall have an outer, support ring diameter of (30 ± 0,1) mm at the diameter of contact with the test piece, and a loading ring diameter of (12 ± 0,1) mm at the diameter of contact with the test piece. The radius of curvature of the specimen contact surface shall be (2 ± 0,2) mm. The jig shall have a means of centring the loading and support rings and the test piece on a common axis to within ± 0,2 mm.

The rings should preferably be made from hardened steel (greater than 500 HV or 40 HRC) in order to minimize damage or roughness caused by the fracturing test pieces.

In order to accommodate slight departures from flatness in the surfaces of the test pieces, a rubber plate, (0,6 ± 0,1) mm thick, of 60 ± 5 Shore hardness shall be placed between the support ring and the test piece, and a piece of paper shall be placed between the test piece and the loading ring.

5.4.2.3 Micrometer in accordance with ISO 3611 capable of measuring to an accuracy of ± 0,01 mm.



See NOTE 2.

Figure 1 — Schematic diagram of biaxial flexural strength test device with concentric loading and support rings

5.4.3 Preparation of test pieces

5.4.3.1 Prepare billets or discs of the test material using methods representative of the production methods of parts for surgery, using the same precursor powder, pressing method and pressure, and firing conditions.

5.4.3.2 The test pieces (see figure 1) shall be circular plates of diameter (36 ± 1,0) mm and thickness (2 ± 0,1) mm. The surface to be tested shall be in a fired state.

5.4.3.3 At least 10 test pieces shall be prepared for determination of mean strength or at least 30 test pieces if a Weibull statistical analysis is required.

5.4.4 Procedure

NOTE 2 This test procedure can also be used for test pieces with other surface treatments not complying with 5.4.3, e.g. ground or polished. In every case the preparation method should be recorded as stated in 5.4.6 b).

5.4.4.1 Measure the diameter of the test piece to the nearest 0,1 mm and the thickness to the nearest 0,05 mm, each in at least three random positions. Calculate the mean diameter and mean thickness.

5.4.4.2 Place the rubber sheet on the support ring of the test jig. Place the test piece on the rubber sheet with the surface to be tested in contact with the rubber and centre it. Place a paper disc on the top of the test piece and place the loading ring on the paper and centre relative to the test piece and support ring.

5.4.4.3 Place the test jig in the test machine and apply a steadily increasing force to the loading ring at a rate of (500 ± 100) $N \cdot s^{-1}$ until the test piece fractures. Record the load at fracture.

5.4.4.4 Inspect the fragments for evidence of the failure origin. If this is more than 0,5 mm outside the inner loading ring, note this fact in the report (5.4.6). For the purposes of calculation of the fracture stress, assume failure within the inner loading ring. Do not discard the result in calculating the mean strength of the test batch.

5.4.4.5 Repeat the test procedure for each test piece in the batch.

5.4.5 Calculation of results

For each test piece, calculate the nominal biaxial fracture stress, σ, in megapascals, as:

$$\sigma = \frac{3F}{2\pi t^2}\left[(1+\nu)\ln\left(\frac{d_s}{d_l}\right) + (1-\nu)\left(\frac{d_s^2 - d_l^2}{2d^2}\right)\right]$$

where

F is the force applied at fracture, in newtons;

t is the mean test piece thickness, in millimetres;

d_s is the mean support ring contact diameter, in millimetres;

d_l is the mean loading ring contact diameter, in millimetres;

d is the mean test piece diameter, in millimetres;

ν is Poisson's ratio, which for the purposes of this test shall be taken as equal to 0,25.

Calculate the mean nominal fracture stress and the standard deviation for the batch of test pieces.

5.4.6 Test report

The test report shall contain the following information:

a) the identity of the ceramic material, details of the batch number and other codes sufficient to identify the test pieces uniquely;

b) the method of preparing the test pieces, including details of the machining procedure used to prepare the test surfaces (see 5.4.3.1);

c) the mean value, the standard deviation and the Weibull statistical data. The position of the apparent site of fracture shall be noted if this falls more than 0,5 mm outside the loading ring diameter.

5.5 Wear resistance

5.5.1 Principle

A ring of alumina ceramic is loaded onto a flat alumina plate, and is rotated through an arc of ± 25° at a frequency of 1 Hz for a given period of time, using water as the surrounding medium. The volume of the wear track on the disc is determined and used as a measure of wear resistance for the purposes of this specification.

5.5.2 Apparatus

5.5.2.1 Ring-on-disc oscillating test device with the capability of positioning the ring test piece concentrically with respect to the disc test piece (figure 2). The ring specimen shall be able to undergo oscillatory rotation about a fixed axis by ± 25° rotation angle at a rate of 1 Hz using a sinusoidal or near-sinusoidal rate of change of angle. The disc-holding device shall include a universal joint to ensure that the

plane of the disc surface coincides with the plane of the ring surface at all times.

5.5.2.2 Profilometric test device, e.g. a skidless diamond stylus instrument, to determine the wear volume removed from the disc test-piece. This device shall allow the wear track cross-sectional area to be calculated from the profile measured.

5.5.3 Preparation of test pieces

5.5.3.1 Prepare billets or discs of the test material using methods representative of the production methods of parts for surgery, using the same precursor powder, pressing method and pressure, and firing conditions. Prepare at least five pairs of test pieces.

5.5.3.2 The test pieces (see figure 3) should have the following dimensions:

Contact surface region of ring test piece:

— inner diameter ($14\ ^{0}_{-0,1}$) mm

— outer diameter ($20\ ^{0}_{-0,1}$) mm

Contact surface of disc test piece:

— diameter $\geqslant$ 25 mm

Other dimensions of test pieces may however be chosen to suit the design of the test device.

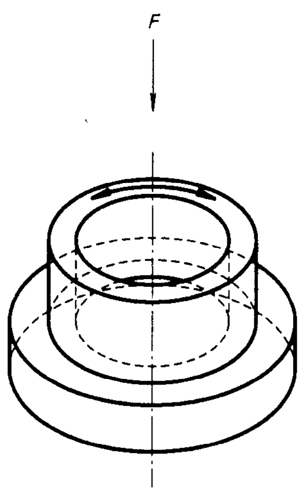


Figure 2 — Schematic diagram of ring on disc wear test

Dimensions in millimetres

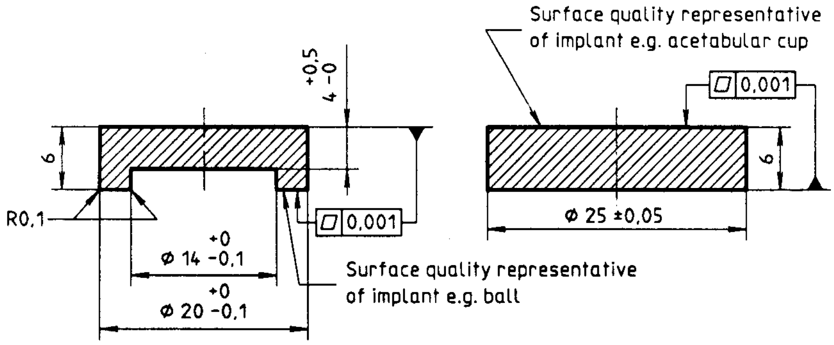


Figure 3 — Geometry of ring and disc test pieces with necessary dimensions defined

5.5.3.3 The contacting surfaces of the test pieces shall be prepared to the following conditions:

Surface roughness, CLA: ⩽ 0,10 µm

Flatness: ⩽ 0,6 µm

The roughness shall be determined in accordance with ISO 468 using a profilometer device having a valid calibration in accordance with procedures in ISO 5436. The flatness shall be measured using an appropriate interferometric device, and the entire contact area of the test piece shall be observed.

5.5.4 Procedure

5.5.4.1 Fix the disc and ring test pieces to the respective holders in the test rig, using a suitable adhesive or clamping device.

5.5.4.2 Apply the following test conditions:

Rotation angle:	± 25°
Axial load:	(1 500 ± 10) N
Frequency:	(1 ± 0,1) Hz
Test time:	(100 ± 1) h
Lubricant:	distilled water
Temperature:	room temperature, with the temperature of the immediate environment of the test pieces monitored and reported.

5.5.4.3 Remove the test pieces from the test device. Clean in distilled water in an ultrasonic tank to remove debris. Dry the test pieces and inspect the worn surfaces. If the wear track in the disc test piece is an incomplete ring, this indicates incomplete contact between the test pieces. Repeat the test with new test pieces.

5.5.4.4 Determine the wear track profile using the profilometer along six symmetrically disposed radial directions commencing 3 mm outside the outer edge of the wear track and finishing 3 mm inside the inner edge of the wear track. Use a sufficiently sensitive scale to delineate the wear track clearly.

5.5.4.5 To determine the departure of the wear track profile from the original surface defined by the profiles on either side of the wear track, proceed as follows. From both sides of the wear track, extrapolate the mean surface profile towards the centre of the track. Using a planimeter or other suitable electronic means, determine the area between the wear track profile and the extrapolated lines to give the wear track cross-sectional area.

NOTE 3 A more sophisticated method is to determine the surface profiles along the same radii before and after the wear test by indexing the test piece in a jig. The wear track area can then be determined by electronic subtraction of one profile from the other.

5.5.5 Calculation of wear volume

5.5.5.1 Determine the average wear track cross-sectional area from the six profiles.

5.5.5.2 Determine the wear volume, V, by multiplying the average wear track cross-sectional area by the average ring length giving:

$$V = \pi(r_o + r_i)A$$

where

A is the mean wear track cross-sectional area, in square millimetres;

r_o is the mean wear track outer radius, in millimetres;

r_i is the mean wear track inner radius, in millimetres.

5.5.6 Test report

The test report shall contain the following information:

a) the method of preparing the test discs, including relationship to the production of parts for surgery (5.5.3.1) and machining procedure adopted;

b) a description of the test apparatus;

c) details of the tests performed to measure surface roughness and flatness of the test pieces;

d) details of the technique used to determine the wear track profile;

e) the stabilized temperature achieved during the operation of the test;

f) the individual values of wear volume from each of five tests, the overall mean value and standard deviation.

(*Continued from second cover*)

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
	IS 1528 (Part 12) : 1974 Methods of sampling and physical tests for refractory material : Part 12 Determination of bulk density (*first revision*)	Technically Equivalent
ISO 5436 : 1985	IS 10707 : 1983 Instruments for measurement of surface roughness by profile methods	do
ASTM C573 : 1986	IS 12107 (Parts 1 to 9) Methods of chemical analysis of alumina-silicate refractory materials	do
ASTM E112 : 1988	IS 4748 : 1988 Methods for estimating average grain size of metals (*first revision*)	do

This Indian Standard has been issued in 15 parts. Other parts of this Indian Standard are:

- Part 1 General requirements
- Part 2 Wrought stainless steel
- Part 3 Unalloyed titanium
- Part 4 Wrought titanium 6-aluminium 4-vanadium alloy
- Part 5 Cobalt-chromium-molybdenum casting alloy
- Part 6 Wrought cobalt-chromium-tungsten-nickel alloy
- Part 7 Wrought cobalt-nickel chromium-molybdenum alloy
- Part 8 Forgeable and cold-formed cobalt-chromium-nickel-molybdenum-iron alloy
- Part 10 Ultra-high molecular weight polyethylene, powder form
- Part 11 Ultra-high molecular weight polyethylene, moulded form
- Part 12 Wrought cobalt-nickel-chromium molybdenum iron alloy
- Part 13 Wrought high nitrogen stainless steel
- Part 14 Wrought titanium 5-aluminium 2,5-iron alloy
- Part 15 Wrought titanium 6-aluminium 7-niobium alloy

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. MHD 2 (2703).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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